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Polychlorinated Biphenyls and Other Organic Chemical Residues in Fish from Major United States Watersheds near the Great Lakes, 1978

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16. ABSTRACT

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FISH, WILDLIFE, AND ESTUARIES

Polychlorinated Biphenyls and Other Organic Chemical Residues in Fish from Major United States Watersheds Near the Great Lakes, 1978

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ABSTRACT

Twenty-six composite samples of fish were collected during 1978 from United States watersheds near the Great Lakes and analyzed for polychlorinated biphenyls (PCBs) and related organic chemicals. PCB mixtures resembling Aroclor 1254 were found in all samples, and mixtures resembling Aroclor 1242 (or 1016) were found in 77 percent of the samples. Total PCB concentrations in the whole-fish composite samples ranged from 0.13 to 14.6 ppm; 65 percent of the samples contained > 2 ppm PCBs. DDT and its metabolites were also found in all samples. ΣDDT concentration was 1.66 ppm, and 81 percent of the samples contained < 1.0 ppm ΣDDT. Chlordane ranged from < 0.001 to 2.57 ppm in 38 percent of the samples. Hexachlorobenzene was found in 65 percent of the samples, ranging from <0.005 to 0.447 ppm. Other chemicals identified by gas chromatography/mass spectrometry included petroleum hydrocarbons and chlorobenzenes, chlorostyrenes, chlorophenols, and chlorinated aliphatic compounds. Fish from the Ashtabula River (Ohio), Rocky River (Ohio), and Wabash River (Indiana) contained extremely complex residues of chlorinated and other organic chemicals.

Introduction

In 1976, authors extended their gas-liquid chromatography/mass spectrometry (GLC/MS) exploratory studies of organic chemical residues in Great Lakes fish to include residues in fish from major United States rivers for the purpose of tabulating polychlorinated biphenyls (PCBs) and other xenobiotic chemicals accumulating in the aquatic environment. A previous work (6) showed that the types and concentrations of chemical residues in fish varied immensely among rivers in the same area of the country. Fish in some rivers in eastern Michigan and Ohio contain PCB residues almost exclusively, whereas fish from the Ashtabula River nearby in Ohio contain at least 19 major chlorinated chemicals in addition to PCBs. Fish from rivers a few miles apart differ in hexachlorobenzene (HCB) residues by a factor of almost 3,000.

The wide variation in both the types and amounts of chemicals in different waters suggests that it is not cost-effective to apply trend-monitoring programs to the problem of determining the extent of contamination by toxic chemicals. Trend monitoring requires a predetermined list of chemicals to monitor, precise methods for measuring small differences in concentration, and, to minimize biological variability, a fairly rigid sampling protocol with respect to species and size. In contrast, the initial problem regarding toxic industrial chemicals is the identification of every major chemical component of residues from hundreds of industrial areas. Such areas may not have diverse fish populations because of the contamination. Finally, order-of-magnitude estimates of residue concentrations by GLC/MS may be used to direct enforcement-related field studies to "hot-spots" for more intensive studies.

A previous work (6) indicated that taking composite samples of any fish near the mouth of a river provides a convenient, enriched sample of bioaccumulable chemicals being discharged into the entire watershed and excludes the bulk of the less persistent, nonaccumulable chemicals attributable to natural products and sanitary wastes. Because the accumulation of chemicals in fish species varies considerably less than the concentration of chemicals in rivers and areas of rivers, composite samples also provide adequate estimates of relative concentrations in the sampling areas. This paper presents the results of exploratory studies of chemicals in fish from rivers in Minnesota, Wisconsin, Indiana, Michigan, Ohio and New York (Figure 1).

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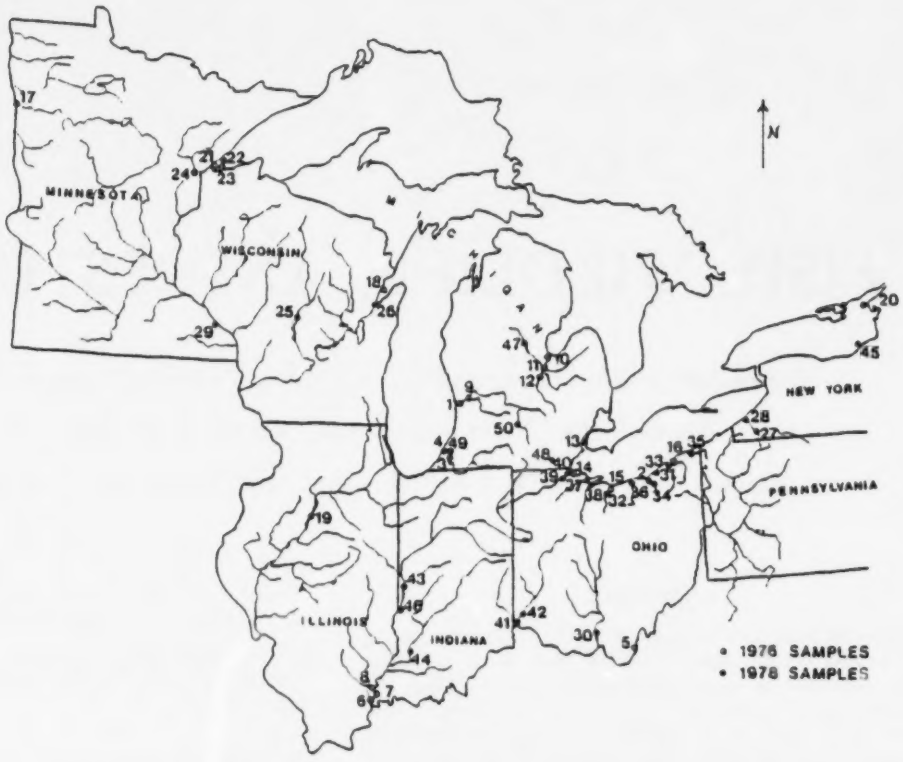

FIGURE 1. *Map of U.S. EPA Region V showing sites where fish samples were collected for GLC/MS analysis of bioaccumulating hazardous organic chemical residues*

Materials and Methods

COLLECTION OF FISH

The areas that were sampled for detailed GLC/MS analyses in 1976 and the 26 sampling areas for the present study (1978) are shown in Figure 1. Table 1 gives a brief description of the 1978 sampling locations.

State and federal field personnel used nets and other conventional apparatus to collect fish. Sampling areas included known problem areas disclosed by previous studies as well as rivers for which little data were available. Where possible, samples were collected near mouths of rivers or confluence of major tributaries. Sample areas are designated as general areas of rivers rather than exact locations because of the migratory nature of many fish and the tendency of bioaccumulable chemicals to contaminate large areas near the discharge. Samples were wrapped in solvent-rinsed aluminum foil, frozen, and shipped with dry ice to the Environmental Research Laboratory in Duluth, Minnesota.

PREPARATION OF SAMPLES

The procedures for preparation and analysis of samples have been described previously (*6*). Composite whole-fish samples were homogenized with a Hobart tissue grinder. Subsamples weighing 20 g were extracted in a Soxhlet extractor with a 1:1 mixture of hexane and methylene chloride. Following Florisil column cleanup, samples were analyzed by electron-capture (EC) and flame-ionization (FI) GLC and multiple-ion-detection (MID) GLC/MS.

GLC analysis provided measurement of PCBs, HCB, DDE, and ΣDDT, as well as information on the complexity of the residue with respect to other nonpolar organic chemicals. MID GLC/MS was used to quantitate *cis*- and *trans*-chlordane, *cis*- and *trans*-nonachlor, heptachlor epoxide, and oxychlordane. A procedural blank was performed with every fifth sample. Duplicate analyses of several samples gave results within 4 percent for both EC GLC and MID GLC/MS. Recovery from laboratory-raised fathead minnows spiked with 1 ppm PCB and 50 ppb pesticides was >92 percent for each compound.

A second subsample (100 g) of each fish was Soxhlet-extracted in a 1:1 mixture of hexane and methylene chloride, cleaned by gel-permeation chromatography (*2*), and qualitatively analyzed by full mass range

TABLE 1. *Concentrations of major chlorinated hydrocarbons in composite whole-fish samples from 22 United States watersheds near the Great Lakes, 1978*

Sampling Area	Sample Composition	PCB Residues, ppm			DDE, ppm	ΣDDT, ppm	HCB, ppb	Chlordane (cis + trans), ppb	Nonachlor (cis + trans), ppb	Heptachlor, ppb	Heptachlor Epoxide, ppb	Oxychlordane, ppb
		1016/1242	1254	Total								
Wisconsin River, below Nekoosa Dam, Wisc.	5 walleye 2 carp 2 white sucker	0.38	0.86	1.24	0.08	0.14	7	•	•	•	•	•
Fox River, below DePere Dam, Wisc.	4 carp 4 white sucker 5 channel catfish	3.61	6.41	10.0	0.75	1.06	18	•	17	•	•	•
Lake Pepin, Mississippi River, Minn.	3 carp	0.15	4.35	4.50	0.36	1.03	37	•	20	•	•	•
Wabash River, below Terre Haute, Ind.	5 channel catfish	0.31	1.77	2.08	0.08	0.15	<5	45	37	27	5	•
Wabash River, above Terre Haute, Ind.	5 carp	0.18	1.07	1.25	0.13	0.21	<5	21	25	•	•	•
White River, Paragon, Ind.	11 channel catfish	0.51	2.65	3.16	0.15	0.47	7	239	288	•	•	•
St. Joseph River, Berrien Springs Dam, Mich.	4 carp	0.34	2.62	2.96	0.35	0.76	9	•	24	•	•	•
Grand River, Clinton County, Mich.	3 carp	<0.10	4.91	4.91	0.56	0.31	36	2,570	3,070	•	•	•
Tittabawassee River, Smith's Crossing, Mich.	5 channel catfish 2 carp	1.25	1.40	2.65	0.23	0.36	64	•	4	•	•	•
Raisin River, Monroe County, Mich.	4 carp	9.83	4.80	14.6	0.25	0.48	14	•	36	•	•	•
Scioto River, Rosemont, Ohio	1 freshwater drum 2 carp 1 channel catfish 1 quillback	0.32	1.98	2.30	0.12	0.40	7	64	125	•	•	•
Ashtabula River, Ashtabula, Ohio	1 white sucker 1 hog sucker 2 stone rollers 1 carp	9.36	1.52	10.9	0.79	0.92	447	•	•	77	•	•
Huron River, Ohio	1 goldfish 2 freshwater drum 2 carp	<0.10	0.62	0.62	0.17	0.26	<5	•	•	8	•	•
Chagrin River, Ohio	3 freshwater drum 2 carp	0.26	3.57	3.83	0.50	0.82	<5	51	434	•	•	•
Rocky River, Ohio	2 carp 1 goldfish	0.84	4.54	5.38	0.52	0.88	21	2,680	1,820	•	•	167
Conneaut River, Ohio	4 freshwater drum 1 carp	<0.10	0.40	0.40	0.04	0.09	39	•	•	•	•	•
Black River, Ohio	5 channel catfish 1 freshwater drum	0.16	1.62	1.78	0.10	0.31	<5	•	•	•	•	•
Portage River, Ohio	5 carp	0.35	2.66	3.01	0.46	0.72	<5	•	•	•	•	•
Sandusky River, Ohio	5 freshwater drum	<0.10	0.59	0.59	0.08	0.15	<5	•	•	•	•	•
Maumee River, Waterville, Ohio	5 carp	<0.10	0.25	0.25	0.02	0.05	<5	25	25	•	•	•
Maumee River, Collen Park, Ohio	3 carp	1.38	3.38	4.76	0.23	0.53	24	•	•	•	•	•
Great Miami River, Miamisburg, Ohio	2 carp 3 goldfish	2.00	5.18	7.18	0.24	0.45	13	24	16	11	•	•
Great Miami River, Elizabethtown, Ohio	3 carp 1 channel catfish	0.78	8.59	9.37	0.66	1.32	13	43	25	•	•	•
Lake Ontario, near Oswego, N.Y.	2 brown trout	1.79	3.97	5.76	1.00	1.66	87	•	•	2	•	1
Cattaragaus Creek, N.Y.	5 white sucker 2 hog sucker 1 brown trout	<0.10	0.13	0.13	0.01	0.03	<5	•	•	•	•	•
Cattaragaus Creek Mouth, N.Y.	3 coho salmon	0.16	1.59	1.75	0.21	0.40	7	•	•	9	•	3

NOTE: • = Below the detection limit of 0.5 ppb.

(m/z 50–550) scanning GLC/MS. Again, procedural blanks were performed with every fifth sample, and spiking experiments showed >90 percent recovery.

ANALYSES

The GLC/MS analyses were performed on a Finnigan 4000 mass spectrometer interfaced to an INCOS data system. Instrument parameters and operating conditions follow:

Column:	glass, capillary, 30-m long by 0.25-mm (ID), coated with SE-30
Temperature, °C:	column programmed from 100 to 225° at 4°/minute (with a 20-minute hold) ion source 280
Carrier gas:	helium flowing at 30 cm/second
Mass scan:	50–550/2 seconds
Electron energy:	70 eV
Emission:	350 mA

MID GLC/MS analyses were also performed on the above equipment, with the same temperature programming, followed by a 20-minute hold. The mass spectrometer was computer-controlled to monitor six ions for equal time during a 2-second period. The six ions were m/z 373 for chlordane, m/z 409 for nonachlor, m/z 272 for heptachlor, m/z 355 for heptachlor epoxide, m/z 387 for oxychlordane, and m/z 442 for defluorotriphenylphosphene (DFTPP).

DFTPP was used as an internal standard for MID quantification. Each extract was spiked with DFTPP to give a concentration of 10 ng/μl. Two standards of the chlordane components of 5 ng/μl and 20 ng/μl each were used as samples after each set of five fish samples. Quantification was based on a 10-ng/μl solution with standard INCOS software. The quantified standards gave values within 4 percent of expected values. The limit of detectability was 0.50 ppb wet weight.

Ion source was operated as above with an emission of 350 mA.

Results and Discussion

PCBs were found in all 26 samples at concentrations ranging from <0.1 ppm in fish from the upper reaches of Cattaragaus Creek, New York, to 14.6 ppm in fish from the Raisin River, Michigan. Aroclor 1254 constituted over 50 percent of the total PCB residues in the majority of the samples. Aroclor 1016/1242 was found in 20 of the 26 samples at concentrations ranging from <0.1 to 9.83 ppm in Raisin River fish. These results are consistent with the authors' previous study of 58 samples, in which Aroclor 1016/1242 was present in 77 percent of the samples (*6*).

Fish from the Fox River (Wisconsin), Raisin River (Michigan), and Ashtabula and Greater Miami Rivers (Ohio) remain heavily contaminated with PCBs. Twelve other samples contained PCB residues >2 ppm. Although PCB concentration in the edible portion of the fish is expected to be lower than that in the whole fish, the present data suggest that 65 percent of the fish samples would pose significant hazards to animals, such as mink, feeding on the fish (*1*).

DDT, once the major organochlorine contaminant in fish in many U.S. waterways, is a minor contaminant in the river systems investigated in the present study. Although DDE was found in all samples, ΣDDT concentration in 81 percent of the samples was below 1.0 ppm; the maximum concentration was 1.66 ppm, in Lake Ontario fish.

HCB was the next most prevalent organochlorine found in the present study; 65 percent of the samples contained measurable quantities. Although most samples contained <0.05 ppm HCB, Ashtabula River fish contained 0.447 ppm. The authors' previous work (*6*) revealed concentrations of 3.14 ppm HCB in fish from the Ashtabula River in 1976. The apparent decline may be the result of sampling variability or of pollution-abatement measures taken since the 1976 discovery. The fact that fish analyzed in the present study were collected upstream from the alleged discharge (*3*) into the Ashtabula River may also be significant. An ongoing field investigation is a direct result and a primary benefit of this type of biomonitoring, because areas of highest contamination are identified for more intensive study at minimum cost.

Chlordane and components of technical chlordane were found in 38 percent of the samples. Although fish from most of the rivers contained <0.05 ppm chlordane, fish from the Grand River, Michigan, and Rocky River, Ohio, contained 2.57 ppm and 2.68 ppm, respectively. The total nonachlor concentrations in these fish were 3.07 ppm and 1.82 ppm, respectively. Heptachlor and heptachlor epoxide were found only in fish from the Wabash, Ashtabula, and Huron Rivers and Lake Ontario. Oxychlordane was present in Rocky River, Lake Ontario, and Lake Erie fish at a maximum concentration of 167 ppb. The coho salmon caught at the mouth of the Cattaragaus Creek, New York, are undoubtedly Lake Erie fish, which would account for the oxychlordane residues.

In addition to chemicals quantified by GLC or MID GLC/MS, the results of exhaustive GLC/MS studies of the fish extracts are summarized in Table 2. The first 25 organic chemicals identified in Table 2 are aliphatic and aromatic hydrocarbons. Heptadecane, pentadecane, and related hydrocarbons are natural products of benthic algae, as well as the results of petroleum contamination from mixtures such as fuel oil. Present methodology permits only qualitative statements about the sources of these compounds, based on FI GLC chromatograms.

Pyridinecarboxamide appeared to be as common as heptadecane in fish from these rivers.

Pentachloroanisole was identified in 15 of the 26 samples of fish. The authors have observed halogenated anisoles in effluents of sewage treatment plants receiving the respective halogenated phenols (*4*). Present information suggests that the anisoles arise from methylation of the corresponding phenols by bacteria. Studies at the Environmental Research Laboratory in Duluth (unpublished data) have shown that fish exposed to halogenated phenols do not produce the anisoles metabolically. The presence of pentachloroanisole may therefore arise from the widespread use of pentachlorophenol as a wood preservative. Because the bioconcentration factor for the methyl derivative of phenols is several orders of magnitude greater than that of the phenol, pentachloroanisole as an environmental contaminant is probably the result of selective bioaccumulation of a more persistent metabolite of pentachlorophenol.

Heptachloronorbornene and hexachloronorbornadiene were found only in the Wabash River fish collected below Terre Haute, Indiana. These two compounds are intermediate chemicals in the production of cyclodiene pesticides, and their occurrence is linked to manufacturing sites for these pesticides. These chemicals were included in Table 2 because they are unique in the Wabash River, not because of widespread occurrence. These data confirm the authors' studies of fish from the lower Wabash (*6*) and suggest a source of contamination in the vicinity of Terre Haute, Indiana. The only other identification of heptachloronorbornene and hexachloronorbornadiene in the aquatic environment was reported by the Food and Drug Administration, U.S. Department of Health and Human Services, in fish from the Mississippi River below Memphis, Tennessee (*5*).

Mirex was identified only in Lake Ontario fish. The compound was first observed in 1973 and was extensively investigated by state and federal agencies.

PCBs were identified in all samples and, although the tetra-, penta-, and hexachlorobiphenyl homologs were predominant, PCBs containing two or three chlorine atoms were found in 19 of the 26 samples.

The above-mentioned quantitative and qualitative data present a reasonably comprehensive description of the chemical residues that contaminate fish in some major rivers. However, these data fail to illustrate adequately the need for improved biomonitoring or analytical methods development. Fish from many of the rivers contain residue mixtures that are similar to the GLC chromatogram of the Maumee River fish presented in Figure 2. This chromatogram was obtained by using a 30-m, wall-coated capillary column and an electron-capture detector. Although there are many chemicals in this extract, PCBs, HCB, and natural products account for all peaks in the chromatogram. Therefore, the residues in this area are comparatively simple to work with, and routine GLC methods should be adequate for any surveillance work.

In contrast, the Ashtabula, Wabash, and Tittabawassee Rivers contain extremely complex mixtures of bioaccumulable chemicals. An electron-capture capillary chromatogram of extract of fish from the Ashtabula River is shown in Figure 3. Chemicals identified include tetra-, penta-, and hexachlorobutadiene; chlorinated benzenes up to hexachlorobenzene; penta- and hexachlorobutylamines; and numerous hexa-, hepta-, and octachlorostyrenes. Despite the identification of almost 100 chemicals in this sample, those identified to date are largely only those in the highest concentrations. A comprehensive study of residues in fish from the Ashtabula and Wabash Rivers is presented elsewhere (*3*).

Even though progress has been made in developing methods for rapid characterization of chemical residues, the long lists of chemicals being published from studies of environmental samples should not lead to the conclusion that present methods are thorough or adequate. In many of the rivers the authors have studied during the past four years, chemicals are present that cannot be identified without improved cleanup methods and instrumental techniques. More important, the number of sample sites studied would have to be increased by an order of magnitude in order to screen even a single sample from industrial areas over the next five years. Major improvements in the current state-of-the-art methods for GLC/MS screening will have to be made before an adequate number of samples can be thoroughly studied.

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TABLE 2. *Chemical contaminants identified by GLC/MS in fish from United States rivers near the Great Lakes, 1978*

Chemical Contaminant	Wisconsin River	Fox River	Lake Pepin	Wabash River (above Terre Haute)	Wabash River (below Terre Haute)	White River	St. Joseph River	Grand River	Tittabawassee River	Raisin River	Scioto River	Ashtabula River
Tridecane	1			2						2		
Tetradecane				2						2		
Pentadecane							2					
Hexadecene			1									
Hexadecane	1		1	1								
Heptadecene							2	2				
Heptadecane				1			1	1	1	1	1	
Octadecane				1							1	
Nonadecane								1				
Eicosane								2				
Naphthalene					1		1			1		
Methylnaphthalene					2	2	2	2		2		
Dimethylnaphthalene					2		2	2		2		
Biphenyl						1						
Methylbiphenyl						2		2		2		
C2-Biphenyl						2		2				
Phenanthrene				2	2	2	2	2		2		
Fluoranthene				2	2	2	2	2		2		
Pyrene					2	2	2	2		2		
Fluorene												
Dibenzofuran												
Acenaphthalene												
Methylbenzanthracene												
Dibenzothiophene												
Pyridinecarboxamide	1	1			1			1	1	1	1	
Terphenyl												
Phenylnaphthalene												
Pentachlorobenzene									1		1	1
Hexachlorobenzene	1	1	1			1	1	1	1	1	1	1
Pentachloroanisole						1	1	1			1	1
Pentachlorophenol					1							
Pentachlorobenzyl alcohol					2							
DDE			1		1	1	1	1	1	1		
TDE			1			1	1	1	1	1		
DDMU			1		1	1	1					
DDT			1		1	1	1	1	1	1		
Heptachloronorbornene					2							
Heptachloronorbornadiene					2							
Mirex												
Photomirex												
Endrin					1							
Monochlorobiphenyl										2		
Dichlorobiphenyl	2									2		
Trichlorobiphenyl	2	2	2		2	2	2	2	2	2		
Tetrachlorobiphenyl	2	2	2	2	2	2	2	2	2	2	2	2
Pentachlorobiphenyl	2	2	2	2	2	2	2	2	2	2	2	2
Hexachlorobiphenyl			2	2	2		2	2	2	2	2	2
Heptachlorobiphenyl			2	2	2	2	2	2	2	2	2	2
Octachlorobiphenyl							2	2	2			
Hexachlorostyrene												2
Heptachlorostyrene												2
Octachlorostyrene												1
Chlordane				1	1	1		1			1	
Nonachlor		1	1	1	1	1	1	1	1	1	1	
Heptachlor					1							1
Heptachlor epoxide					1							
Oxychlordane												

NOTE: 1 = Confirmed by GLC and MS retention time data; 2 = confirmed by MS data; 3 = suggested by MS data.

TABLE 2 (cont'd.).

Huron River	Chagrin River	Rocky River	Conneaut River	Black River	Portage River	Sandusky River	Maumee River (Waterville)	Maumee River (Collen)	Great Miami River (Miamisburg)	Great Miami River (Elizabethtown)	Lake Ontario	Cattaraugus Creek	Cattaraugus Creek (Mouth)
		2			2								
		2			2								
											2		
	2	1	1							1	1	1	1
1	1	1	1		1	1	1			1			
1			1							1			
1			1				1			1			
			2							2			
		2	2	2									
		2	2										
		1		1									
								2		2			
		2	2	2						2			
2		2	2	2	2				2				2
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				2					2				
		3	3	3									
		1	1					1	1	1	1		
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1	1	1	1	1	1				1	1			1
1		1	1		1				1				1
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1	1	1	1	1	1				1	1			1
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	2	2		2	2		2	2	2	2	2		2
2	2	2	2	2	2	2	2	2	2	2	2	2	2
2	2	2	2	2	2	2	2	2	2	2	2	2	2
2	2	2	2	2	2	2	2	2	2	2	2	2	2
2	2	2	2	2	2			2	2	2	2		2
	1	1					1		1	1			
	1	1					1		1	1			
									1		1		1
		1											

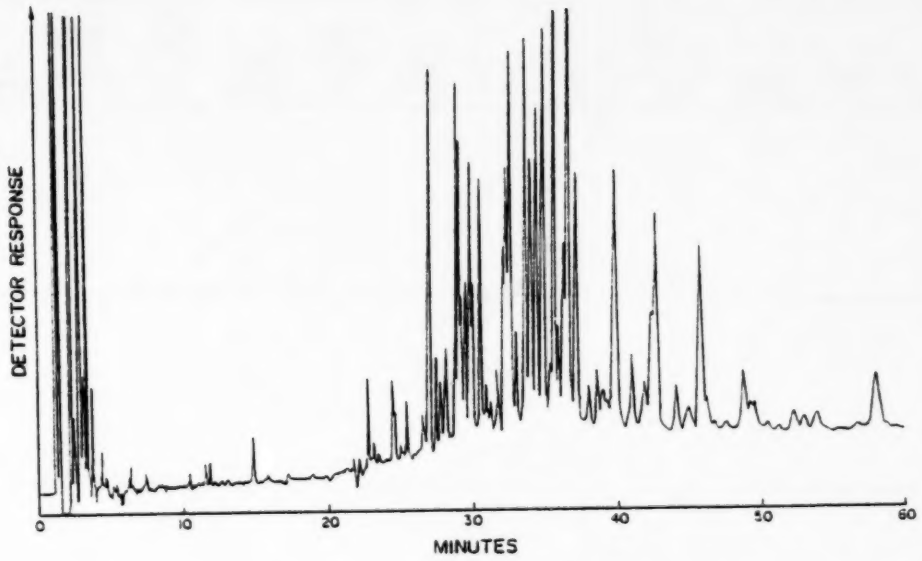

FIGURE 2. *Capillary electron-capture chromatogram of fish extract from Maumee River, Ohio.*

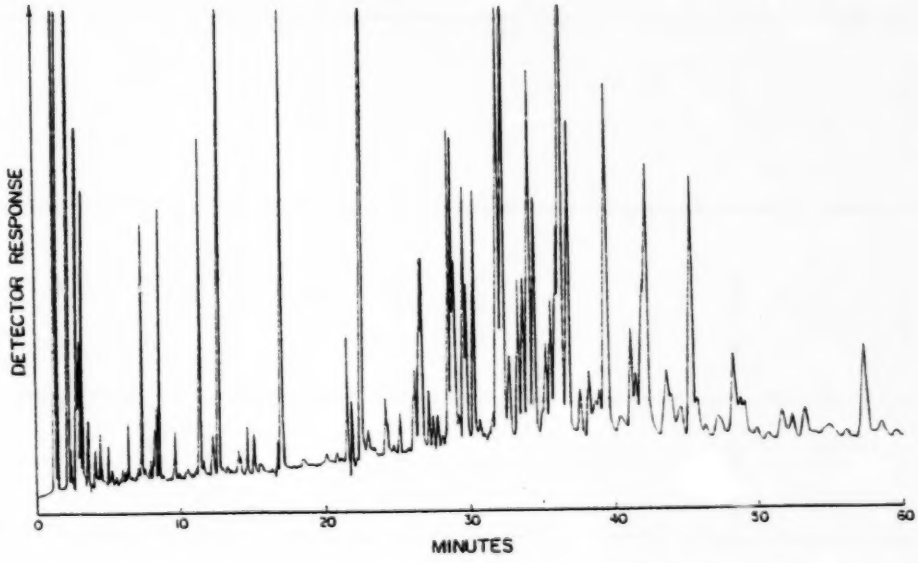

FIGURE 3. *Capillary electron-capture chromatogram of fish extract from Ashtabula River, Ohio.*